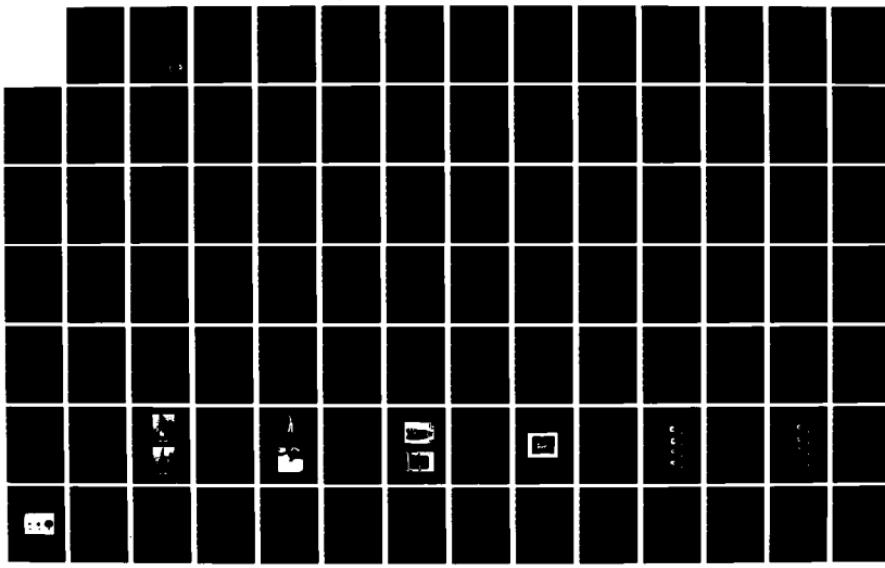


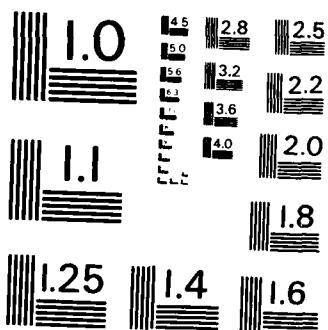
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IN VITRO STUDY COMPARING THE MARGINAL LEAKAGE OF DIFFERENT  
POWDER/LIQUID RATIOS OF ZOE

By

Dennis James Zent  
MAJ, DC, M.S.D.  
1985, pages 91  
Indiana University School of Dentistry

Indianapolis, Indiana

This study assessed the marginal leakage of three powder/liquid ratios of a temporary cement (ZOE), which was inserted into simulated endodontic access preparations placed in tooth structure, as well as in amalgam and composite resin.

Class V cavity preparations with a 90° cavosurface angle were made in 110 anterior and bicuspid extracted human teeth. Thirty of these teeth were set aside to await placement of the temporary mixes. Forty of the teeth were restored with amalgam and the remaining 40 with composite resin. Ten each from the amalgam and composite resin groups served as controls. All specimens were aged two weeks before the temporary cement was placed.

All 30 experimental teeth in each of the three groups had simulated access preparations placed into tooth structure or through the restorative material. In each

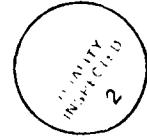
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group, 10 teeth were then restored with a thin ZOE mix, 10 with a medium mix, and 10 with a thick mix.

All specimens were stored in tap water at 37° C except for necessary manipulation. All were subjected to the stress of 2500 thermocycles between two water baths with a temperature differential of 40° C and 30-second immersion time in each bath. Each cement mix was tested for marginal leakage two weeks after placement. Marginal penetration was assessed using radioactive <sup>45</sup>Ca generated autoradiographs. Each autoradiograph was given a leakage classification under multiple, coded, blind conditions by two evaluators.

Statistical analysis using ridit mean and standard deviation for each experimental group was performed with multiple comparisons using the Neuman-Keul Test and analysis of variance to determine significant differences ( $p<.05$ ) in leakage values. There was a significant difference in marginal leakage of the thick and medium mixes placed in composite resin when compared to the thin mix ( $p<.01$ ). Of the restorative material used, amalgam demonstrated significantly greater marginal leakage than the composite resin ( $p<.01$ ).

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IN VITRO STUDY COMPARING THE MARGINAL  
LEAKAGE OF DIFFERENT POWDER/LIQUID  
RATIOS OF ZOE

by

Dennis James Zent

Submitted to the faculty of the Graduate School  
in partial fulfillment of the requirements for  
the degree of Master of Science in Dentistry,  
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## INTRODUCTION

The ability to seal the margins of an access preparation with a temporary restorative material has become an important consideration in endodontics. Since multiple endodontic appointments are common, a temporary cement that prevents the exchange of fluid between the root canal system and mouth is required.

1

Weine suggests that the ideal temporary cement should maintain the degree of asepsis obtained during the canal bioinstrumentation. This goal of canal asepsis is a basic principle of the endodontic triad and has been supported by 2 studies which relate a higher percentage of success when 3-6 canal asepsis is maintained between appointments.

7

Smith reported that dental cements have generally been employed as sealing agents, and that the varied demands imposed on temporary cements have been fulfilled by only zinc phosphate and zinc oxide and eugenol cements.

8-11

Other studies suggest that no interappointment temporary restorative material completely seals the cavity 12 preparation. Going stated that the interface of tooth structure and restorative material is not impenetrable but is a dynamic microcrevice which allows the passage of ions and molecules. This phenomenon is known as micoleakage and

is affected by many factors which may lend to either an increase or decrease in marginal leakage. These factors include the permeability of the enamel wall, manipulative variables and the physical properties of the restorative material.

13-19

Many studies have investigated microleakage at the interface of the restorative material and tooth structure.

20-23

Additional studies have dealt with marginal leakage as it applies to interappointment temporary restorations in endodontics. Marosky and associates compared the sealing ability of available products. These cements were tested for use as temporary restorations during endodontic therapy and their microleakage was compared. The premixed cements produced the best seal. Of the hand mixed cements, only zinc oxide and eugenol provided an adequate interappointment seal.

23

Recently, Orahood showed that zinc oxide and eugenol had both the best and worst leakage pattern. A possible explanation is that the mixes were not consistent, since the powder to liquid ratios and mixing time were not standardized. Both Marosky and Orahood suggested a need for additional studies to evaluate marginal leakage with emphasis on standardization of working time and powder to liquid ratios. Controlling these variables should provide useful information concerning the potential of obtaining a

predicatable marginal seal with this material. Use of the superior ratio that provides a superior seal would be helpful in maintaining interappointment canal asepsis.

The purpose of this study was to investigate the marginal microleakage that occurs at the interface of zinc oxide and eugenol cement, mixed with different powder/liquid ratios, against tooth surface and two commonly used restorative materials, alloy and composite resin. A <sup>45</sup> Ca autoradiographic technique was used to evaluate the specimens for marginal leakage after the restorations had been exposed to water storage and thermocycling.

## REVIEW OF LITERATURE

## MARGINAL LEAKAGE

Marginal microleakage is a complex phenomenon that has perplexed the dental profession. This leakage around dental restorations has been related to hypersensitivity, marginal discoloration, recurrent caries, and interappointment complications in endodontic therapy. <sup>12</sup> Going stated that although ionic charge and chemical reactivity of diffusing fluids have a part in marginal leakage, the physical and chemical nature of the restorative materials and the clinical skills of the operator play equally important roles. A number of techniques have been used to evaluate marginal leakage, and are described here under separate headings.

### Air Pressure

Air pressure has been used by several investigators to evaluate marginal leakage. <sup>24-26</sup> <sup>24</sup> Harper demonstrated marginal leakage of air bubbles at the margins of artificial cavity restorations placed in a steel die and restored with amalgam. The die was submerged in water and bubbles were observed when air pressure was applied to the floor of the restoration.

Steel dies with amalgam were subjected to air pressure  
25 by Hollenback, who found that after being immersed in  
120° F water and then cooled to room temperature, all  
amalgam specimens leaked.

26 Pickard and Gayford chose air pressure as a medium for seeking out the paths of leakage. They used amalgam restorations placed over a zinc phosphate cement base in class V cavity preparations. It was thought that this method could expose actual defects of continuity without obliterating a nearby small leak. Because paths of different sizes have different critical pressures and can be viewed from different angles, the process of observation can be selective. Compressed air was passed through a needle into the pulp chamber and into contact with the floor of the restorations. A light microscope was used to observe the submerged, non-thermocycled specimens. All specimens displayed a pattern of leakage which decreased with time. The obvious limitation to this technique would be a false negative as a result of air flow blockage by debris in the pulp chamber.

#### Artificial Caries

27 28 Grieve and Kidd used an artificial caries technique to evaluate marginal leakage. Acidified gelatin was used to create in vitro artificial secondary caries-like lesions in

extracted teeth. Subsequent examination of ground sections of the teeth by polarized light microscopy showed the extent of artificial caries around the restorations. This technique can be performed at normal oral temperatures and has clinical relevance since it relates enamel dissolution and leakage.

#### Bacterial Penetration

Bacterial penetration has been used to study permeability at the margins of dental restorations. In 1929

<sup>29</sup>

Fraser used small glass ampules to test the ability of bacteria to penetrate certain dental cements and filling materials. Bacteria and culture medium were placed in a glass ampule separated by various cements and alloys.

Leakage was determined if the sterile culture medium sealed by the materials became cloudy and demonstrated the organism used to seed the external surface. The specimens were not subjected to thermocycling and revealed marginal leakage in 5 of 21 restorations tested.

<sup>30</sup>

In 1955 Seltzer evaluated the marginal leakage around acrylic resin and amalgam restorations placed in class V cavity preparations in freshly extracted teeth. The teeth were placed in thermocycled and non-thermocycled groups. The thermocycled specimens were subjected to 30 second temperature changes one or two times a day from 7 to 60 days. The restorations were then subjected to bacterial penetration,

after which the restorations were removed and dentin shaving from the cavity preparations were placed in broth containing culture tubes. Since color-producing microorganisms were used, marginal leakage was demonstrated by a reddish growth on an agar slant streaked with inoculum from a cloudy culture tube. A distinct relationship was demonstrated between an increased number of thermocycles and a corresponding increase in marginal leakage. It must be kept in mind that inadvertent contamination may be an artifact and distort the results.

31

Rose and associates used microorganisms to test the marginal leakage of resin materials placed in class V cavity preparations in freshly extracted teeth. Limited thermocycling was performed on the specimens. Upon removal of the restorations, culture tubes containing sterile broth were inoculated with dentin shavings from the walls of the cavity preparations. Marginal leakage occurred in 60 per cent of the specimens.

32

Likewise, Mortensen and others in 1963 used microorganisms to evaluate the marginal leakage of four materials. Restorations placed in extracted teeth were thermocycled and exposed to bacterial penetration. All four materials showed leakage. Recurrent decay is associated with marginal leakage and bacterial penetration, which gives these studies a clinical relevance, but the disadvan-

tage is that the results do not determine the degree of leakage of each specimen.

#### Conductimetric

<sup>33</sup>

Jacobsen and von Fraunhofer reported in 1975 on a conductimetric technique to evaluate leakage. Non-conductive materials, such as resins and silicate cements, can be tested in an artificial cavity. The floor of the cavity was formed by a nickel plated brass electrode and the walls were formed by glass tubing. Lactic acid was used as the electrolytic marginal penetration solution. Both thermocycled and non-thermocycled specimens were tested. The cavity wall restoration interface was incorporated into an electrochemical cell, and the changes in the current passing through this cell reflect changes in the dimensions of the interface gap space.

A similar technique was used by Jacobson and von

<sup>34</sup>

Fraunhofer in 1976 to check for marginal penetration in extracted teeth that had been endodontically treated. The apical seal of vertically condensed gutta percha was challenged by a penetrating solution of one per cent potassium chloride. When the solution penetrated the apical seal and reached a steel rod placed into the coronal end of the canal through an occlusal opening, corrosion of the steel was established and was measured for quantity and rate. The

authors said that the technique suggested a quantitative means of measuring apical sealing procedures.

35

In 1983 Mattison and von Fraunhofer determined the microleakage of a number of endodontic sealers. Extracted teeth were treated and sealed with sealer without a solid core. Microleakage was measured over a period of 30 days by the flow of electric current as a result of the penetration of a potassium chloride solution at the margin of the tooth sealer interface.

#### Dyes

Organic dyes have been used extensively to determine marginal leakage. In 1939 Grossman used dye solutions, saliva colored with dye, and bacteria to test the hermetic sealing properties of ten temporary filling materials. He used glass capillary tubes which permitted direct visual examination.

36

Buchanan in 1951 used a solution of methylene blue dye and microscopic evaluation to study the leakage of silicate and resin restorations. His testing method was similar to that used by Grossman (capillary tubes).

37

38

39

Massler and Ostrovsky and Weiss tested various restorative materials for leakage with methylene blue dye. Leakage was determined when the dye penetrated around the margins of the restorative materials, placed in glass tubes,

and stained a cotton fiber wick. The test permitted a good evaluation of the rate of marginal leakage.

40

41

Hirsch and Weinreb and Parris and Kaspsimalis used a two per cent aqueous solution of aniline blue dye to test the marginal leakage of dental restorations placed in freshly extracted human teeth.

42

Trail and Sausen in 1962 attempted to measure dye penetration at the margins of class V cavities placed in extracted human teeth and restored with zinc phosphate cement. The chemical and physical properties of various dyes were evaluated to determine which dyes were most suitable for use in leakage studies. The authors recommended the following eight: basic fuchsin, safranine O, Malachite green, methylene blue, methyl violet, crystal violet, auramin O, and prontosil rubrum soluble.

43

In 1966 Christen and Mitchell placed class V restorations in bovine teeth and subjected them to fluorescein and rhodamine B dyes. The teeth were sectioned after varying lengths of time and viewed under ultraviolet light. The investigators reported that fluorescein showed marginal leakage satisfactorily and held the most promise for future research since it is inexpensive, permits reproducible results, requires short immersion periods and contrasts sharply with the natural fluorescence of teeth. Additionally, the dye is non-toxic, easy to photograph, and adaptable to

clinical as well as laboratory investigations. It was noted that ZOE affects the fluorescence of these dyes and therefore would be unsatisfactory for evaluating marginal leakage of ZOE cements.

44

In 1968 Gross used class V preparations in the teeth of hamsters to study microleakage *in vivo*. Amalgam restorations were placed in the preparations without cavity liners or varnish. The test teeth were isolated with a rubber dam and flooded with a two per cent solution of fluorescein dye for five minutes. The teeth were then extracted, sectioned, evaluated and compared to control teeth which were restored *in vitro* and subjected to the same dye. The author suggested that the increased microleakage observed *in vitro* when compared to the *in vivo* specimens is valid.

45

Loiselle et al. in 1969 used fluorescein dye for a study of microleakage in class V cavity preparations, placed in bilaterally paired mandibular teeth scheduled for extraction, and restored with amalgam without cavity liners or varnish. The patients returned for dye penetration tests and removal of the teeth after a two week wait. The results showed massive leakage around the restorations *in vitro* and relatively little leakage *in vivo*. The authors expressed the view that marginal microleakage should be determined *in vivo* if clinical practice conditions are to be approximated.

<sup>46</sup>

Going stated that although the fluorescent dye method is a significant contribution for the evaluation of in vivo marginal leakage, it is of questionable value in terms of scientific validity because it fails to provide a quantitative method for assessing marginal microleakage around dental restorations.

#### Neutron Activation

The technique of neutron activation analysis technique used in vitro and in vivo to quantitatively evaluate marginal leakage was described in 1968 by Going et al.<sup>47</sup> Cavity preparations were placed in 120 human teeth, 108 in vitro and 12 in vivo. The preparations were restored with 10 different restorative materials. The teeth were then soaked in a non-radioactive solution of manganese nitrate (in vivo specimens were extracted after exposure to the solution), placed in the core of a nuclear reactor and exposed to a pulsed neutron flux, recovered, and sectioned, with gamma-ray emission then being measured quantitatively. The non-radioactive manganese solution diffuses into the teeth and is converted into radioactive <sup>56</sup>Mn inside the nuclear reactor. Gamma-ray emission of <sup>56</sup>Mn was measured by a scintillation detector linked to a 400 channel gamma-ray spectrometer and data in the form of numerical printouts were converted into total counts by plotting a graph of counts versus channel numbers. This not only reflected

the total distribution of the tracer label within the area of the restoration, but also offered a means of testing the marginal seal of restorations placed *in vivo* for varying periods. The distribution of manganese determined in these studies suggests that considerable penetration of the underlying dentin and possibly pulp, occurs as a result of marginal leakage. Microleakage was greater *in vivo* than *in vitro*, but it should be mentioned that the *in vitro* specimens were not subjected to thermocycling.

48

In 1974 Meyer et al. discovered an improved method of neutron activation analysis of microleakage. Since the presence of manganese, either in the tooth or restoration, causes a variability of the results, the authors suggested an alternative tracer. It was determined that dysprosium was a more suitable tracer; it provided the least variation in the results and allowed the fastest activation and counting procedures. A major limitation of the technique used in both of these studies is that the researcher must have access to a nuclear reactor.

#### Light Microscope

49

Nelson, Wolcott, and Paffenbuger used a light microscope in 1952 to view the opening and closing of the margins of restorations placed in extracted teeth and subjected to temperature changes. They viewed fluid exchange along the

margins of eight different filling materials. The restored teeth were immersed in ice water for 30 seconds, then removed, dried, and viewed under a binocular microscope. All specimens of each material extruded fluid at the margins and the authors assumed that this marginal percolation was caused in part by a difference in the coefficients of thermal expansion of the tooth and the restoration and by thermal expansion of the fluid occupying the crevice between the tooth and the restoration. In addition to the exchange of fluids resulting from thermal changes, other chemical and physical forces may augment this seepage or act independently in causing it. These other forces may include capillarity, dialysis, diffusion, and changes in hydraulic or gas pressures. Since the limit of visual acuity is approximately 50 microns, and since it seems reasonable to assume that a channel 10 microns in diameter could develop at the junction of restoration and tooth, the light microscopic method of examination has definite limitations. These limitations become apparent when even the authors note that micro-organisms, acids, and enzymes commonly attributed to the production of dental caries are smaller than 10 microns. Therefore any unseen defect of 10 microns in diameter is already a large cavity and this unseen defect would allow molecules of acids and enzymes to be washed into and out of this space. The authors concluded that in light of the

current concepts of the mechanisms of dental caries, marginal percolation may be an explanation for the recurrence of caries at the margins of some restorations.

#### Scanning Electron Microscope

The scanning electron microscope has made it possible to observe marginal adaptation with both great magnification and depth of field. Boyde and Knight in 1969 and Lee and Swartz in 1970 reported examining tooth specimens directly. Unfortunately the vacuum processes of coating and observing the specimens can cause artificial cracking and gap formation due to differential shrinkage of the enamel, dentin and restorative material.

Grundy in 1971 and Barnes in 1979 advocated replication techniques which would also allow continued evaluations of in vivo specimens since the tooth is not extracted. The authors supported the use of this replication technique for intra-oral studies of surfaces of teeth and restorations when qualitative assessment is required.

#### Radioactive Isotopes

The rationale and methodology involved with the use of radioactive tracers was defined by Bartelstone in 1950. He said that usefulness of an isotope as a tracer depended upon its availability in suitable form, its ability to be

incorporated into the material to be studied, and its availability for recovery and measurement. An additional consideration is the ability of the alpha and beta particles to produce an image on photographic film. Disadvantages of the tracer technique were cited as: the radiation of the isotope, its half-life, the cost and availability of a particular isotope, and the special training of personnel and special laboratory facilities necessitated by the safety hazards in handling radioactive materials.

56

In 1951 Wainwright reported the penetration of enamel defects by a radioactive solution of <sup>45</sup>Ca. He used a laboratory monitor to count radioactivity and autoradiographs to record results. Intact enamel was not penetrated, although there was an uptake on the surface of enamel and cementum which was not removed by washing.

57

In the same year, Armstrong and Simon investigated the penetration of a radioactive solution of <sup>45</sup>Ca into class V restorations placed in extracted bicuspids. Using autoradiographs, the authors reported that <sup>45</sup>Ca penetrated the margins of all filling materials in varying amounts.

58

Wainwright reported in 1953 that radioactive isotopes were being used to evaluate marginal leakage at many dental research facilities. The isotope ions can penetrate a smaller gap and to a greater depth than dyes, and can be detected in minute amounts.

59

In 1959 Swartz refined and standarized the radio-isotope technique to allow for screening materials and evaluating manipulative techniques. Procedures such as sealing the teeth, determining isotope exposure time, and washing and sectioning the teeth were defined and established.

60

Going et al. in 1960 compared a variety of isotopes to determine whether ionic change and chemical reactivity enhanced marginal penetration. Metallic restorations showed a marked adsorption of negatively charged isotopes  $^{131}\text{I}$ ,  $^{35}\text{S}$ ,  $^{32}\text{P}$ , and the positively charged ions  $^{22}\text{Na}$  and  $^{86}\text{Rb}$  showed little or no surface adsorption. Additionally  $^{45}\text{Ca}$  and  $^{35}\text{S}$  showed selective and deep penetration into marginal defects and produced the clearest and sharpest autoradiographs. The authors concluded that the depth of marginal penetration of an isotope is influenced not only by its ionic charge and chemical reactivity but also by the physical and chemical nature of the restorative material.

61

In the same year Going and associates used crystal violet dye and  $^{131}\text{I}$  tracer to test the marginal integrity of all filling materials in common clinical use. Both old and fresh restorations were studied. All restorations showed some degree of penetration by  $^{131}\text{I}$ .

62

In 1961 Jeffay characterized the three types of particles emitted during the disintegration of an unstable

isotope. The alpha particle is slow-moving, has little penetrating ability but possesses great ionizing power. The beta particles travel near the speed of light and have moderate penetrating and ionizing power. Gamma particles have no mass, travel at the speed of light, and although they do not tend to ionize material, they will penetrate most substances. Jeffay stated that for the most part beta particle emitters such as <sup>14</sup> C, <sup>35</sup> S and <sup>45</sup> Ca are useful as research tools, and gamma emitters like <sup>131</sup> I and <sup>60</sup> Co are <sup>45</sup> more useful for diagnostic and therapeutic purposes. <sup>45</sup> Ca is a low energy beta particle and will penetrate fewer layers of cells than a high-energy beta particle. This makes <sup>45</sup> Ca well suited for autoradiographic studies.

Swartz and Phillips in 1961, utilizing the autoradiographic technique refined and standardized by Swartz two years earlier, conducted in vitro studies on the marginal leakage of six different restorative materials. The relative sealing abilities of the restorations were determined on the basis of the marginal permeability to <sup>63</sup> Ca. It was the same basic technique previously used by Armstrong and <sup>57</sup> Simon, <sup>64</sup> Sausen et al. and <sup>65</sup> Crawford and Larson, with modifications. The concentration of the radioactive solution of calcium chloride was 0.1 millicurie per milliliter and the pH was adjusted to 5.5 by adding dilute sodium hydroxide. The root surfaces and root canals were sealed

with a combination of fingernail polish and tin foil. Only the margins of the restorations were exposed to the isotope. The teeth were then immersed in the isotope solution for two hours, rinsed in running water for one hour, and then scrubbed with a detergent and water. After the tin foil was removed, the roots were again thoroughly scrubbed. Longitudinal sections were prepared through the restorations using a wet 400 mesh carborundum wheel. The pulp was removed from the teeth and the cut surfaces were carefully brushed and dried before becoming placed on x-ray film for preparation of the autoradiographs. The x-ray film was exposed to the specimens for 17 hours, processed and evaluated for leakage.

The radioisotope technique of evaluating microleakage has become one of the principal methods used in recent years. In several studies <sup>63,66-68</sup> the in vitro results using the autoradiographic technique correlated well with the in vivo results. This supported the in vitro conditions used to stress the specimens prior to exposure to the radioisotope solution: (1) storage in water at 37° C and (2) thermocycling.

67

In 1976 Peterson et al. reported that subjecting restored teeth to thermal stress mimics the in vivo ingestion of hot and cold foods. This can be accomplished by using a thermocycling machine to immerse the specimens in two separate constant temperature baths. The specimens are

subjected to a 30° C differential. Using thermocouples placed in restored cavity preparations of extracted teeth, and immersed in water baths, these investigators were able to determine the time required for the temperature change to be transmitted through the restoration. The water bath temperature was transmitted to the cavity floor within 15 seconds. An immersion time of 30 seconds assured that the restoration and tooth would reach the water bath temperature.  
45

Ca autoradiographic technique is relatively simple and requires no complex equipment. As with any in vitro test, applicability to the in vivo situation may be questionable.  
68

However, McCurdy and associates reported that the autoradiographic technique produced similar results in parallel in vitro and in vivo studies.  
69

In 1969 O'Brien and associates questioned the ability of the autoradiographic technique to determine a non-leaking system. Later research by Guzman and associates indicates that the technique can discriminate between leakage and non-leakage. A disadvantage of the technique is that the data are essentially qualitative since it is difficult to measure the amounts of leakage. Recent refinements of the evaluation procedure, using multiple blind reading by two evaluators and ridit analysis of the data, have enabled quantitative statistical analysis of the results.  
18,19,71,72  
73

## RESTORATIVE MATERIALS MARGINAL LEAKAGE

46

Going in 1972 noted in a review of microleakage around dental restorations that the marginal permeability at the interface between tooth and restoration has been studied many times. These studies emphasized that the margins of restorations are not fixed, inert and impenetrable borders, but rather dynamic microcrevices with a busy traffic of ions and molecules. The author said that microleakage around restorations appears to be a series of phenomena and not a single entity. Although ionic charge and chemical reactivity of diffusing fluids have a part in marginal leakage, the clinical skills of the operator and the physical and chemical properties of the restorative material play important roles.

## Silver Amalgam

38

In 1954 Massler and Ostrovsky reported on the sealing properties of a number of commonly used filling materials under controlled conditions. The materials were placed in glass tubes and subjected to gentian violet or methylene blue. Dye penetration was noted when a cotton wick that was packed above the filling began to stain. Zinc oxide eugenol and amalgam showed the most effective sealing qualities. The authors acknowledged that the conditions did not duplicate the clinical situation.

63

In 1961 Swartz and Phillips reported the results of in vitro studies on the marginal leakage of restorative materials. Six restorative materials (amalgam, silicate cement, zinc phosphate cement, and three types of resin) were placed in extracted teeth and their sealing abilities were determined by marginal permeability of  $^{45}\text{Ca}$ . The restorations were subjected to the isotope solution after different time intervals of 24 hours, one month and six months. The margins of the 24-hour-old amalgam restorations were readily penetrated, but leakage diminished with aging of the restoration. Use of a cavity varnish improved the marginal seal of the restorations. Some leakage was observed with all silicate and zinc phosphate cement restorations. Leakage increased when the restorations were subjected to thermal changes and the magnitude appeared to be related to temperature differentials.

66

45

In the same year, Phillips et al. again used  $^{45}\text{Ca}$  to evaluate the marginal leakage and the influence of aging on amalgam, silicate cement, zinc phosphate cement and resin. Restorations were placed in humans and dogs. The restorations in dogs were tested with the isotope after 48 hours, 30 days, 60 days and six months. Intervals of one week, one month and three months were used for the humans. The behavior of silicate restorations was variable. Zinc phosphate cement showed leakage which diminished with age

and the resin materials effected a relatively good seal for the short period involved in the study. Additionally, the study showed that initially the margins of amalgam restorations were readily penetrated by the isotope, but leakage appeared to diminish as the restoration aged. Use of a cavity varnish apparently improved the initial sealing ability of amalgam.

74

Roydhouse and Weiss in 1967 investigated leakage around the margins of silicate cement, acrylic resin, and amalgam with cavity varnish. Their results showed that leakage around restorations was a series of phenomena and not a single entity. Laboratory tests of leakage are influenced by the operators, the nature of the tracers used, and the nature of the sections examined. Also, the fluid's ability to penetrate depends upon the pressure differences between the external surface and the internal dentinal face. Additionally, it was discovered that the sealing qualities of cavity varnish may diminish as it thickens. Several thin layers of cavity varnish are preferable to one thick layer.

Marginal leakage of dental restorations subjected to thermal stress was investigated by Guzman and associates in 1969. They reported on microleakage of four dental materials (amalgam, silicate cement, resin and reinforced epoxide resin) placed in class V cavity preparations of extracted human anterior and bicuspid teeth. The effect

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of aging and the influence of a cavity varnish on amalgam restorations were also investigated. Microleakage around one-week amalgam restorations, measured with radioisotope, increased when the restorations were subjected to 500 temperature cycles. Three-month specimens showed good sealing ability that was not affected by thermal stress. Amalgam restorations, placed into cavity preparations coated with a varnish, showed little or no marginal penetration, and thermocycling did not impair the sealing qualities of these restorations. The remaining restorations sealed the cavities quite effectively over the study period.

75

Hembree and Molinary in 1974 reported that leakage patterns around amalgam restorations are not affected by varying the mercury content. However, use of a cavity varnish minimizes the initial leakage. The authors state that a cavity varnish is important to reduce the leakage around an amalgam for the first several days until a reduction in leakage can occur due to a deposition of corrosion products from the amalgam.

76

In 1976, Phillips stated that a changing pattern of operative dentistry was emerging due to advances in biomaterials. Advances in the biologic characteristics of materials and new methods resulted in reduced microleakage. New systems of amalgam alloy were fostered by identification of the gamma two phase as the phase which lacks corrosion

resistance. The resulting corrosion-resistant dispersed phase and high copper alloys provide a superior restorative material.

77-82

Numerous studies have reported that corrosion-resistant high copper alloys exhibit leakage. This leakage was reduced when cavity varnish was used before the amalgam restoration was placed.

Dental amalgams make up approximately 80 per cent of  
83 all single-tooth restorations. One reason for the more than 160 million amalgam restorations placed each year is its outstanding record of clinical performance. When these restorations are properly inserted, marginal leakage decreases with age, apparently due to corrosion products formed on the restorations at the tooth-restoration interface. Use of a cavity varnish is necessary to seal this interface until the accumulation of corrosion products, which is somewhat slower in the newer high copper alloys, takes place in sufficient amounts to seal the margins.

#### Composite Resin

84

Phillips states in his text that most composite  
85 resins are based on the formulation of Bowen. Composite resin has three components, the first being a resin matrix (BIS-GMA). Inorganic particles (glass or silica), better known as a filler, make up the second component. A coupling

agent which bonds the first two components together and also acts as a stress absorber at the filler-resin interface is the third component of composite resin.

Improved esthetics, rapid polymerization and the convenience of a paste-type material make composite resin the material of choice for class III, IV and V restorations. The technique will undoubtedly become more popular with the innovation of the acid etching technique coupled with use of an unfilled resin as a bonding agent, as described by

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Phillips. He recommended a 30-50 per cent concentration of phosphoric acid for etching. Combining this approach with use of a resin with low viscosity (usually an unfilled BIS-GMA resin) as a liner before inserting the composite resin allows wetting of the tooth. This in turn assures penetration into the etched area to form retentive tags and improved marginal sealing.

86

In 1976 Eriksen and Buonocore reported that marginal leakage was reduced when composite restorations were extended onto peripheral etched enamel, with or without application of a bonding agent to the cavity preparation.

87

In the following year, Forsten reported decreased marginal leakage using an intermediary resin before placing the composite in the previously etched preparation.

88-95

Subsequent studies have all reported that margin leakage is reduced or eliminated when the cavosurface enamel

is etched and the cavity preparation is primed with an unfilled resin before a composite restoration is placed.

95            96            18  
Additionally, Crim and Mattingly, Crim and Porte reported that beveling the cavosurface margin before etching the peripheral enamel helped reduce marginal leakage.

#### ENDODONTIC INTERAPPOINTMENT RESTORATIVE MATERIALS

Dental cements have generally been used as sealing agents, and Smith<sup>7</sup> has described cements as the most important materials used in general practice today. During endodontic procedures, temporary filling materials are used to seal the access opening in the crown between treatments to prevent fluid exchange between the root canal and the mouth. A prime consideration in the selecting of a temporary filling material is it's ability to seal the margins of the cavity within which it is placed. The authors of endodontic textbooks agree on the importance of sealing the access opening between appointments, but they can not agree on the material to be used.

36  
Grossman in 1939 said that the temporary materials used for sealing root canals needed further study. Since he considered a bacteria-tight seal was a necessity during root canal treatment, he investigated the permeability to dyes, to saliva and to test organisms, of the more commonly used temporary filling materials. The temporary filling materials

were condensed into glass tubing to a thickness of 2-3 mm and then challenged by dyes, saliva or test organisms. Marginal leakage was noted when cotton fibers on the opposite side of the cement were stained. The zinc oxide eugenol cements appeared to seal the best, under all test conditions.

Gutta percha leaked moderately and zinc phosphate cement demonstrated the worst leakage.

39

Weiss tested the sealing properties of a number of commonly used filling materials. His 1958 study showed that at room temperature, zinc oxide eugenol cement provided an excellent marginal seal.

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In 1960 Parris and Kapsimalis tested the marginal leakage of nine filling materials placed in the access openings of recently extracted non-carious, non-restored anterior human teeth. The root apex of each tooth was restored with amalgam to prevent apical leakage of the dye. The restorative materials were placed over cotton fibers and allowed to set for 24 hours. Twenty-seven specimens were tested at room temperature and the rest were tested after 10 thermocycles between 60°C and 4°C. Aniline dye was used to test marginal leakage. Staining of the cotton fibers indicated marginal leakage. Zinc oxide eugenol cement, Cavit and amalgam demonstrated no leakage at room temperature. Cavit demonstrated no leakage after thermocycling, whereas half of the zinc oxide eugenol specimens showed leakage with thermal stress.

In 1964 Parris et al. tested the effect of temperature change on the sealing properties of various filling materials. In their view, previous studies testing the sealing properties of temporary filling materials had not been evaluated under conditions similar to those encountered in the mouth. Since one prime consideration in selecting a temporary filling material is its ability to seal the margins of the cavity in which it is placed, the authors compared the sealing ability of temporary filling materials under conditions simulating those found in the mouth. In the first part of the study they used the dye penetration technique described previously, including room temperature and thermocycled specimens. The temporary filling materials that did not permit dye penetration were Cavit and Kwikseal. In the second part of the study restorations of the test material were placed in the apex and access openings with cotton fibers in the pulp chamber containing a culture medium. The specimens were thermocycled through a bacterial solution at both 4° and 60° C. The cotton fibers were recovered and placed in culture tubes with broth which was then streaked on agar plates. Cavit, Kwikseal, Kalsogen and ZOE did not permit bacterial penetration. A third part of the study was conducted to determine whether the temporary filling materials, in themselves would inhibit bacterial growth. ZOE, Dentin,

Kwikseal, Kalsogen and Cavit produced the most pronounced inhibitory effect. Only Cavit and Kwikseal maintained a leakproof cavity in both dye and bacterial studies.

<sup>98</sup>

In 1969 Tagger and Tagger reported on the marginal leakage of zinc oxide eugenol and Cavidentin. Class V cavity preparations 1.5 mm deep were placed on the facial and lingual surfaces of 43 extracted human teeth. One preparation was restored with ZOE and the other preparation, on the same specimen, was restored with Cavidentin. The specimens were then challenged by one of three different dyes for 72 hours. The results indicated ZOE resisted penetration at the margins of one of the three dyes. Cavidentin resisted marginal penetration of the dyes, but the material itself was permeated by the dyes.

<sup>99</sup>

In 1976 Ingle explained that all faulty restorations and caries must be removed and replaced with a temporary filling material to prevent bacterial contamination from salivary leakage, as well as the percolation of intracanal medication. Because of its ease of manipulation and its excellent sealing properties, he considered Cavit the filling material of choice for temporary closure of access cavities. The only drawback cited was its slow setting property, requiring one hour in the mouth to reach a complete set.

<sup>100</sup>

In the following year, Krakow and co-workers reported on an in vivo study of temporary filling materials

used in endodontics in anterior teeth. The same anterior tooth in each of the subjects was used for all seven materials tested, although the number of tests for each material varied due to lack of patient compliance. The tests were performed on teeth which had previously been treated endodontically. At the time of treatment, radiographic examination had shown a periapical area of rarefaction which had subsequently healed. A sterile cotton pellet, moistened with phosphate buffer, was placed in the pulp chamber and the chamber was sealed with the temporary filling material being tested. After a minimum of one week the cotton pellets were removed and cultured anaerobically. Each positive culture was streaked on agar plates and an estimate of viable organisms originally present was made from the colony count. On the basis of the quantity of microorganisms grown anaerobically, differentiation was made between no leakage, minor leakage and gross leakage. The possibility of antibacterial action of the filling materials themselves was investigated in the second part of the study. Findings with Cavit and Cavitron showed no or minor leakage in 27 of 32 cases and ZOE showed no leakage in 6 of 7 cases. The assessment of antibacterial action of the filling materials showed that ZOE had the greatest antibacterial action.

22

Marosky and associates in 1977 assessed the marginal leakage of temporary sealing materials used between endo-

dontic appointments. The materials tested in this in vitro study were: Temp-Seal, Cavit, ZOE, IRM, zinc phosphate cement and polycarboxylate cement. Extracted human anterior and premolar teeth with restorations placed in access cavity preparations were divided into groups and tests were conducted three and 10 days after placement of the restora-  
45 tions, using calcium chloride Ca as the tracer. For each time interval there were thermocycled and non-thermoncycled specimens. The 10-day group was subjected to 2500 thermo- cycles, compared to 500 for the three day group. For each material at each interval, marginal leakage increased as the thermocycling increased. Temp-Seal showed superior sealing ability followed closely by Cavit. ZOE showed good sealing ability and placed third.

101 In the same year Olmstead and associates evaluated the surface softening of Cavit, IRM and zinc phosphate cement when in contact with endodontic medicaments (formocresol, camphorated parachlorophenol and metacresylacetate). Changes in surface hardness were evaluated with a Knoop hardness tester. IRM was softened considerably by the medicaments which had no effect on Cavit or zinc phosphate cement.

102 In 1979 Maerki et al. reported that the effectiveness of materials in preventing the ingress and egress of salivary ions and bacteria is dependent upon numerous physical and mechanical factors. Test specimens of ZOE, IRM, Cavit and

gutta percha were packed into 4 by 8 mm cylindrical molds and stored at 37° C and 100 percent relative humidity for 24 hours before further handling. Each specimen was subjected to compressive testing on a constant strain-rate testing machine. Relaxation measurements were conducted within a constant temperature water bath at temperatures ranging from 11-56° C. This range is consistent with temperatures found within the oral cavity. The results indicate that within the vicinity of ambient mouth temperature, the relaxation characteristics of an unmodified ZOE cement were more favorable than those of Cavit or IRM, which is a resin modified ZOE.

103

According to Grossman, the adequacy or inadequacy of temporary filling materials for sealing access cavities has not been given the consideration it deserves. A material used to seal the endodontic access opening during the time between patient appointments must provide a hermetic seal. Temporary filling materials should: (1) be impervious to bacteria and fluids of the mouth, (2) hermetically seal the cavity peripherally, (3) place no pressure on the medication, (4) set in minutes after insertion, (5) withstand forces of mastication, (6) be easy to manipulate, (7) be easy to remove and (8) harmonize with the color of the tooth structure. Grossman reported that ZOE produced the best hermetic seal, though Cavit was acceptable for anterior teeth.

In 1982 Berstein and Todd investigated the marginal leakage of IRM, ZOE, Cavit and amalgam, tested in vitro without thermocycling. The researchers used 40 extracted, single rooted human teeth to make access preparations and removed the root canal contents. Access preparations were made 6 mm deep to allow an adequate bulk of material for testing. The teeth were separated into four groups of 10 and restored with the sealing agents. Within each group, five teeth were allowed to set for five minutes and the other five set for 24 hours. After the setting period the teeth were subjected to a 0.1 per cent solution of rhodamine B dye for 48 hours, removed and sectioned at a depth of 1, 3 and 5 mm from the access opening. The sections were viewed at 5x under ultraviolet light and leakage was recorded if the dye could be seen or fluorescence detected at the tooth-material interface. The five-minute setting time revealed that IRM had significantly more leakage than Cavit, ZOE or amalgam. There was no significant difference between any of the materials at the 24-hour setting time.

<sup>23</sup> Orahood in 1984 conducted an in vitro study on marginal leakage between temporary sealing materials and restorative materials. He investigated the marginal leakage of Cavit and ZOE inserted into simulated endodontic access preparations placed through two commonly used restorative materials, amalgam and composite resin. Class V cavity

preparations were placed in 60 anterior and bicuspid extracted human teeth. Thirty were restored with amalgam and 30 with composite resin. The specimens were stored in water for two weeks prior to placement of either Cavit or ZOE. Ten teeth from each group served as controls; the remaining teeth from each group had simulated access openings placed through the restorative material and were then restored with either Cavit or ZOE. All specimens were stored at 37° C and subjected to 2500 thermal cycles with a 40° C temperature differential. Each cement was tested for marginal leakage, using radioactive <sup>45</sup>Ca generated autoradiographs, two weeks after placement. There was no significant difference in marginal leakage of Cavit or ZOE, regardless of which restorative material they were adapted against (p<.05).

According to radioisotope studies, both amalgam and composite resin are effective restorative materials. Unfortunately, they would be difficult and time-consuming to use at the conclusion of each endodontic appointment. Additionally, a new access preparation would be necessary at subsequent visits. Weine states that zinc oxide and eugenol cement provides an excellent seal and is much easier to remove than resin or amalgam. He further stated that Cavit and Cavit G could be used, but as long as ZOE is available it is the material of choice.

METHODS AND MATERIALS

Zinc oxide and eugenol cement is one of the most effective materials known for a temporary filling. It has been widely used during endodontic treatment as an interappointment temporary restoration and is excellent for minimizing microleakage.

Three powder-to-liquid ratios of zinc oxide and eugenol cement (Temrex, Interstate Dental Co., New Hyde, NY) were tested for their sealing ability at the interface of tooth surface and two commonly used restorative materials: silver amalgam (Tytin, S.S. White, Philadelphia, PA), and a direct filling composite resin (Concise, 3M Co., St Paul, MN).

A total of 110 extracted, noncarious, nonrestored human anterior and premolar teeth were obtained from oral surgeons in private practice. The teeth were cleaned and stored in tap water prior to the start of the study. Care was taken to guard them against dehydration during storage and manipulation.

The 110 teeth were randomly divided into three groups: Groups I and II, 40 teeth each; and Group III, 30 teeth. Group I teeth were restored with silver amalgam alloy, and Group II teeth with composite resin. Group III required no restorations and were placed in tap water storage. Groups I

and II were divided into four subgroups of 10 specimens each. Subgroup A of each group served as the control and therefore had no simulated endodontic access opening. Subgroups B of each group had a simulated access preparation placed through the restorative material, which was then restored with ZOE mix #1. Subgroup C of each group had a simulated endodontic access preparation placed through the restorative material, which was then restored with ZOE mix #2. Subgroup D was prepared in the same way and restored with ZOE mix #3.

Cavity preparations for the restorations were placed in the facial surfaces of each tooth with a number 57 tungsten carbide bur operated at high speed with water spray coolant. The teeth were hand held and prepared using a bur speed of approximately 325,000 R.P.M. Each bur was used to prepare no more than 10 cavities. Conventional 90° butt-joint cavosurface margins were placed so that the axial wall extended to a depth of approximately 3.0 mm. Code numbers were randomly assigned and cut into the root surface of each tooth.

The 40 teeth assigned to Group I were restored with silver amalgam and the 40 teeth assigned to Group II were restored with composite resin (these techniques will be described below). Group III was to have its simulated endodontic access preparations cut directly into the tooth structure and therefore was left in tap water storage at

this time. There was a four-week interval between the time when the silver amalgam and composite resin restorations were placed and the start of the marginal leakage test. All specimens were stored in tap water in a controlled temperature oven ( $37^{\circ}\text{ C}$ ) during this four week interval. Two weeks elapsed between placement of the restorative materials and the time when the simulated endodontic access preparations were cut and restored with temporary cements. Thus the time lapse from placement of the cements in all three groups to the start of the marginal leakage test was two weeks. During the two weeks following placement of the cement restorations the specimens were subjected to thermocycling. Upon completion of the thermocycles and the two week storage, the marginal leakage was tested. The two-week storage period simulated an acceptable interappointment period as described by Weine.<sup>2</sup>

#### Temporary Cement Mixes

A pilot study was conducted to determine the powder to liquid ratio of the zinc oxide and eugenol mixes to be used. The volume of liquid was set at 0.2 ml of eugenol. Powder was added until a mix of the consistency commonly used to seal an access preparation was obtained. Two additional mixes, one thicker and the other much thinner, were obtained by adjusting the amount of powder. The final powder/liquid ratios for each mix are listed in Table I.

Numerous trial mixes were attempted before the standard mixing time was set at two minutes. A slump test was then performed on each of the mixes to compare their flow. Each ratio was mixed for two minutes, then 1 ml of the cement was loaded on a glass slide, covered with a second slide and subjected to a 15 kg load for five minutes. The total time elapsed from the start of mixing until the cement was subjected to the test load was three minutes.

#### **Amalgam Restorations**

Compressed air was used to dry the teeth in Group I upon completion of the cavity preparation, and two coats of cavity varnish (Copelite, H.J. Bosworth, Skokie, IL) were placed. The amalgam, prepared according to manufacturer's directions, was placed into the cavity by hand condensation, carved and burnished. The specimens were stored in tap water immediately after placement of the restorations. On day following insertion of the amalgam, the restorations were finished using the Sof-lex disc series (Sof-lex Discs, 3M Co., St Paul, MN). The specimens were stored in tap water at 37° C.

After the two-week storage in water the 30 teeth of Subgroups B, C, and D had simulated endodontic access preparations placed within the amalgam restorations. The simulated preparations penetrated through the restoration

and into the dentin of the pulpal wall, extending to a depth of 3.0 to 4.0 mm (Figure 1). The preparations were cut with a number 57 tungsten carbide bur operated at high speed (approximately 325,000 R.P.M.) with water spray coolant. The teeth were hand-held.

#### **Amalgam-ZOE Mix #1**

Simulated endodontic access preparations for Subgroup B were dried with compressed air and restored with ZOE mix #1 (Figure 2), consisting of 1.0 gram of powder and 0.2 ml of eugenol mixed for two minutes using a glass slab and cement spatula. The ZOE powder was weighed on a analytical balance with accuracy maintained to the nearest milligram. The eugenol was measured using a 1.0 cc syringe. The cement was placed into the cavity with the blade end of a Tarno FP hand instrument and the cavity was filled flush with the margin of the simulated endodontic access preparation cut in the amalgam restoration. The condenser end of the instrument was moistened with water, and used to condense the material. The blade end was used to smooth the margins of the restoration. The zinc oxide and eugenol cement was allowed to bench-set for three minutes before the specimen was placed in tap water in a controlled temperature oven held at 37° C. Specimens of Subgroup B were kept in storage for two weeks following insertion of ZOE mix #1, before proceeding with the marginal leakage test.

**Amalgam-ZOE Mix #2**

The simulated endodontic access preparations of Subgroup C were restored with ZOE mix #2, (0.750 grams of powder and 0.2 ml of eugenol). The cement was placed by the method of manipulation previously described. Upon completion of the ZOE mix #2 restorations, and following the three-minute bench setting time, the teeth of Subgroup C were stored in tap water held at constant temperature for two weeks.

**Amalgam-ZOE Mix #3**

The simulated endodontic access preparations of Subgroup D were restored with ZOE mix #3 (0.500 gram of powder and 0.2 ml of eugenol). The cement was placed by the method of manipulation previously described. Again, upon completion of the ZOE mix #3 restorations, and following the three minute bench setting time, the teeth of Subgroup D were stored in tap water held at constant temperature for two weeks.

**Composite Resin**

The teeth in Group II were restored with composite resin. The cavity preparations were completed as described previously. Upon completion, each of these 40 teeth had a

45° bevel placed around the cavosurface margin of the cavity with a number 169 bur. The cavity preparations were then etched for one minute with a 37% unbuffered phosphoric acid solution supplied by the manufacturer of the composite resin (Concise Etching Liquid, 3M Co., St Paul, MN). A cotton pellet was soaked with the acid solution and applied to the cavity preparation. The surface was kept moist throughout the etching period using a dabbing motion. The tooth was then flushed with water for one minute and dried with compressed air.

The composite resin was mixed and placed according to manufacturer's directions. Bonding resin (Concise Enamel Bond, 3M Co., St Paul, MN) was applied to the cavity walls prior to the insertion of the composite resin into the resin-primed cavity. The composite resin was then allowed to bench harden. Fifteen minutes later the restoration was finished to the cavosurface margins with Sol-lex discs. The specimens were immediately stored in tap water for two weeks at 37° C.

The Group II teeth were then randomly divided into four subgroups of 10 each. Subgroup A consisted of control specimens and thus remained in storage without further treatment. The 30 teeth of Subgroups B, C and D had simulated endodontic access preparations placed within the composite resin restorations. As described previously, the simulated endodontic access preparation penetrated through

the composite resin restoration into dentin. The bur, bur speed, and water coolant were the same as described for amalgam restorations.

#### **Composite Resin - ZOE Mix #1**

The simulated endodontic access preparations of Subgroup B were restored with ZOE mix #1, consisting of 1.000 gram of powder and 0.2 ml of eugenol mixed by the method of manipulation previously described. Upon completion of the ZOE mix #1 restorations, and following the three-minute bench-setting time, the teeth of Subgroup B were stored in tap water held at 37° C for two weeks.

#### **Composite Resin - ZOE Mix #2**

The simulated endodontic access preparations of Subgroup C were restored with ZOE mix #2 (0.750 gram of powder and 0.2 ml of eugenol mixed by the method of manipulation previously described). Upon completion of the ZOE mix #2 restorations, and following the three-minute bench-setting time, the teeth of Subgroup C were stored in tap water held at 37° C for two weeks.

#### **Composite Resin - ZOE Mix #3**

The simulated endodontic access preparations of Subgroup D were then restored with ZOE mix #3 (0.500 gram of

powder and 0.2 ml of eugenol mixed by the method of manipulation previously described). Upon completion of the ZOE mix #3 restorations, and following the three-minute bench-setting time, the teeth of Subgroup D were stored in tap water in the controlled temperature oven at 37° C for two weeks.

#### Nonrestored Teeth

Group III consisted of 30 nonrestored, noncarious teeth which had been stored in tap water since extraction. The teeth were randomly divided into three subgroups of 10 each. Each tooth in Subgroups A, B and C had a simulated endodontic access preparation placed in the facial surface with a number 57 tungsten carbide bur operated at high speed with water spray coolant. The teeth were hand-held and prepared with a bur speed of approximately 325,000 R.P.M. Each bur was used to prepare no more than 10 simulated endodontic access preparations. Conventional 90° butt-joint cavosurface margins were placed so that the axial wall extended to a depth of approximately 3.0 mm. A code number was assigned and cut into the root surface of each tooth.

#### Nonrestored Teeth - ZOE Mix #1

Simulated endodontic access preparations for Subgroup A were dried with compressed air and restored with ZOE mix #1

(1.000 gram of powder and 0.2 cc of eugenol, mixed by the method of manipulation previously described). Upon completion of the ZOE mix #1 restorations, and following the three-minute bench-setting time, the teeth of Subgroup A were stored in tap water held at 37° C for two weeks.

#### **Nonrestored Teeth - ZOE Mix #2**

The simulated endodontic access preparations of Subgroup B were restored with ZOE mix #2 (0.750 gram of powder and 0.2 cc of eugenol mixed by the method of manipulation previously described). Upon completion of the ZOE mix #2 restorations, and following the three-minute bench-setting time, the teeth of Subgroup B were stored in tap water held at 37° C for two weeks.

#### **Nonrestored Teeth - ZOE Mix #3**

The simulated endodontic access preparations of Subgroup C were restored with ZOE mix #3 (0.500 gram of powder and 0.2 cc of eugenol mixed by the method of manipulation previously described). Upon completion of the ZOE mix #3 restorations, and following the three-minute bench-setting time, the teeth of Subgroup C were stored in tap water in the controlled temperature oven at 37° C for two weeks.

### Thermocycling

All specimens of Groups I, II and III were subjected to thermal stress during the two-week storage time following placement of the ZOE cement restorations and prior to leakage tests. The teeth were thermocycled for a total of 2500 times between two water baths, having a temperature differential of 40° C. The hot water bath was maintained at 45° C and the cold water bath at 5° C. The immersion time in each bath was 30 seconds, with a transfer time between baths of ten seconds. Upon completion of the thermocycling procedure, the teeth were again placed in the 37° C temperature oven to await the end of the two-week storage period.

### <sup>45</sup>Ca Autoradiographic Technique

This procedure was standarized by Swartz<sup>59</sup> in 1959, and is used regularly in leakage studies at Indiana University School of Dentistry.

Each tooth was painted with fingernail polish except for the immediate area of the restoration. While the polish was still tacky, a layer of tin foil .001 inch thick was used to cover the entire root surface of the tooth (Figure 3). The tooth was again painted with fingernail polish, ensuring that the edges of the foil were sealed but

avoiding the immediate area of the restorations. This was done to prevent isotope from entering the root canal from the apex.

The wrapped specimen was allowed to dry for one hour and was inspected to ensure that the restoration and immediate surrounding tooth structure had not been inadvertently covered with fingernail polish.

Each group of teeth was then submerged in a solution of  $\text{CaCl}_2$  for two hours at room temperature. The solution was of a 0.1 millicurie per milliliter concentration with a pH adjusted to 5.5 by addition of 0.1 N sodium hydroxide.

The pH was adjusted to decrease the possibility of enamel decalcification due to the pH of the  $\text{CaCl}_2$  alone. Next, the teeth were rinsed under running tap water for one hour and then scrubbed with a soft tooth brush and a detergent solution for five minutes. The tin foil was removed and the teeth rescrubbed with brush and detergent for five minutes. This was done to rid the teeth of any excess isotope.

The teeth were then ground on one proximal surface with a 400 grit silicon carbide wheel (Figure 4) with continuous water irrigation until a flat surface was produced which contained the cross section of the restorations (Figure 5). Care was taken to ensure a smooth and flat surface for better contact between the tooth and film. The pulpal tissues were debrided and the teeth were again scrubbed with brush and

detergent for five minutes each, rinsed, and thoroughly dried to prevent moisture contamination of the film emulsion. In a darkroom with a safelight, each prepared section was placed ground side down on the emulsion of an ultraspeed dental x-ray film (Eastman Kodak Co., Rochester, NY) secured to a 2x2x1/8 inch Plexiglass slab and held in place with a rubber band (Figure 6). The specimen code number was then penciled onto the corner of the film. This unit was then wrapped in tinfoil (Figure 7) and kept in a light proof container for approximately 17 hours. After exposure of the autoradiograph, the teeth were removed and the film was developed in an automatic film processor.

#### Microleakage - Evaluation

The test autoradiographs were evaluated independently by two examiners experienced in reading autoradiographs. The examination procedure was standardized in the following manner. One autoradiograph representative of each leakage category was labeled, mounted and available for comparison during the evaluation process. One set of four autoradiographs served as the standard against which the extent of penetration of the restorative materials was scored (Figure 8). Any autoradiograph equal to Figure 8a was given a value of "1" and was considered to have no leakage. Any autoradiograph falling between Figure 8a and 8b was considered

to have slight leakage and was assigned a value of "2." Any autoradiograph that had more leakage than the example in Figure 8b but less than Figure 8c was considered to have moderate leakage and was assigned a value of "3." Autoradiographs showing more leakage than Figure 8c were considered to have severe leakage and were assigned a value of "4." Figure 8d represents an autoradiograph consistent with a value of "4."

Leakage at the margins of the temporary cement mixes (ZOE #1-3) was compared to a second set of four autoradiographs which served as the standard for isotope penetration of the temporary cement mixes (Figure 9). The values applied were labeled "1" through "4" and were identical to those used in Figure 8.

Each examiner performed three separate film readings, with at least 24 hours elapsing between evaluations, recording isotope penetration of both the restorative materials and the different temporary cement mixes. The examinations were conducted using the same lighting and magnification each time. The coded specimens were available for comparison with the coded autoradiographs when necessary. Numerical values were assigned in the manner described for Figure 8. A single film reading was obtained by using the film readings which agreed. For example, if an examiner had readings of 1,2, and 1, the single value used for that film would be 1.

#### **Intraevaluator-Interevaluator Correlation**

Intraevaluator correlation was tested using the three film readings provided by both examiners for 25 randomly selected specimens. Correlation was tested using Pearson's correlation coefficient (Appendix A). Interevaluator correlation was verified by testing 25 randomly selected film reading values of one evaluator against the film reading of the other evaluator (Appendix A).

#### **Statistical Analysis**

Statistical analysis using ridit analysis to determine the population ridit values, ridit mean, and standard deviation of each experimental group was performed after assignment of values. Computer analysis of the data with multiple comparisons using Neuman-Keul's Test and analysis of variance (Program-Anavar, CDC-6600 Computer: Indiana University-Purdue University at Indianapolis) was performed to compare the leakage between experimental groups.

## RESULTS

The discs formed in the slump tests performed on the three temporary cement mixes (ZOE #1,2,3) are shown in Figure 10. The mean diameters of the disks formed by the various mixes are recorded in Table I.

Mix #1 was extremely viscous and appeared nearly set upon application of the test load. Its flow barely allowed it to overcome the shape it had assumed upon placement between the glass slides. Average diameter of the three specimens was 14 mm.

Mix #2 flowed evenly upon application of the test load and the disk formed was nearly circular. Average diameter of the three specimens was 22 mm.

Mix #3 was extremely fluid and flowed readily when the load was applied. The mix quickly spread over the glass slide and in some areas flowed over the sides. Average diameter of the disks was 50 mm.

With respect to evaluation of the leakage test, the intraevaluator and interevaluator correlation using Pearson's correlation coefficient (Appendix A) showed no significant difference in either case (Table II). The ridit value, ridit mean, and standard deviation were obtained for each group using the leakage values determined by the examiners.

Leakage values of the simulated endodontic access preparations that were placed through amalgam, through composite resin, and in tooth structure and then restored with each of the three temporary cement mixes are shown in Table III and Figure 11.

The greatest leakage of all occurred in the amalgam specimens when the access preparation was restored with the thin mix (#3). However, statistical analysis showed that differences in leakage among the three mixes were not significant ( $p > 0.05$ ). Likewise, in the composite resin specimens greater leakage occurred when the access preparation was restored with the thin mix (#3). There was a significant difference ( $p < .01$ ) between the thick mix (#1) and the thin mix (#3), and between the medium mix (#2) and the thin mix (#3) at the .01 probability level. On the other hand, no significant difference in leakage was seen between the thick mix and the medium mix (#1-2).

Among the preparations placed in tooth structure, the greatest leakage occurred with the medium mix (#2). There was no significant difference in leakage values between any of the mixes ( $p > 0.05$ ).

During the experiment three teeth were lost to cracking, as shown in Table III, which accounts for the fact that three of the study groups consisted of only nine teeth, rather than the original 10.

Overall, as can be seen in Table IV, the best seal was observed when the access preparation was placed through composite resin and restored with the medium mix (#2). The greatest leakage was observed when the access preparation was placed through amalgam and restored with the thin mix (#3).

Leakage data for the restorative materials demonstrated that an amalgam restoration had significantly greater marginal leakage than a composite resin placed in an acid etched, resin primed, beveled cavity preparation (Table V and Figure 12).

FIGURES AND TABLES

FIGURE 1. Simulated endodontic access preparation.  
The cavity preparation was placed  
through the restorative material into  
dentin.

FIGURE 2. Temporary restorative material. The  
simulated endodontic access preparation  
was restored with temporary restorative  
cement.

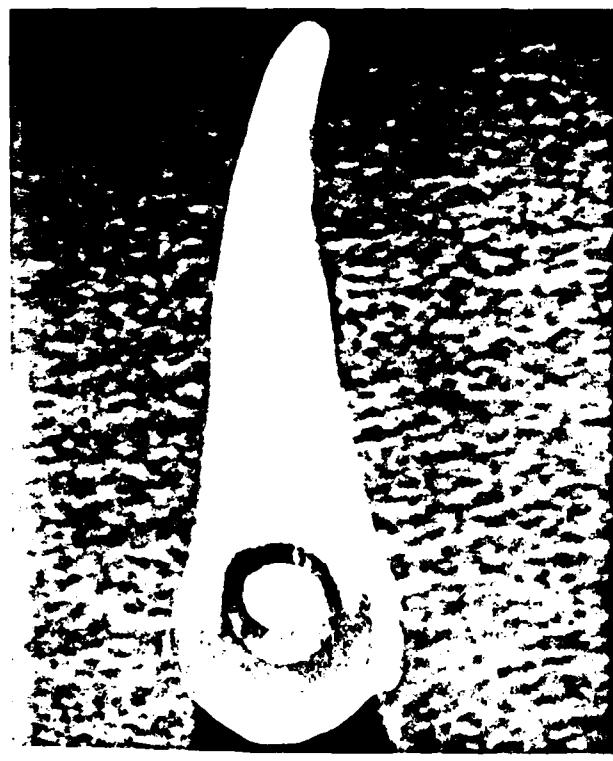
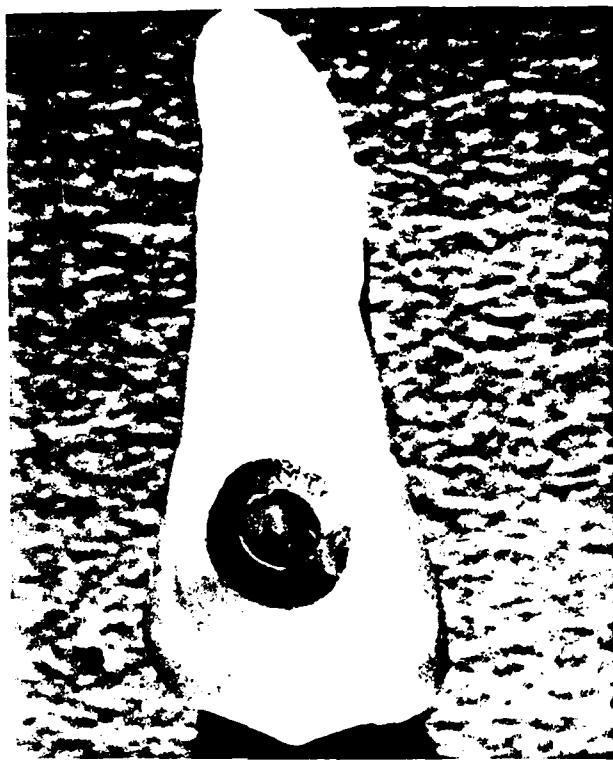


FIGURE 3. Sealed specimen. Specimen sealed with fingernail polish and tin foil, in preparation for  $^{45}\text{CaCl}_2$  immersion.

FIGURE 4. Specimen sectioned. Each specimen was sectioned on a horizontal rotary silicone carbide wheel.



FIGURE 5. Sectioned specimen. Each specimen was sectioned until a flat surface was produced which contained a cross section of the restoration.

FIGURE 6. Film exposed to radioisotope. The sectioned specimen was placed on a dental x-ray film mounted on a plexiglass slab.

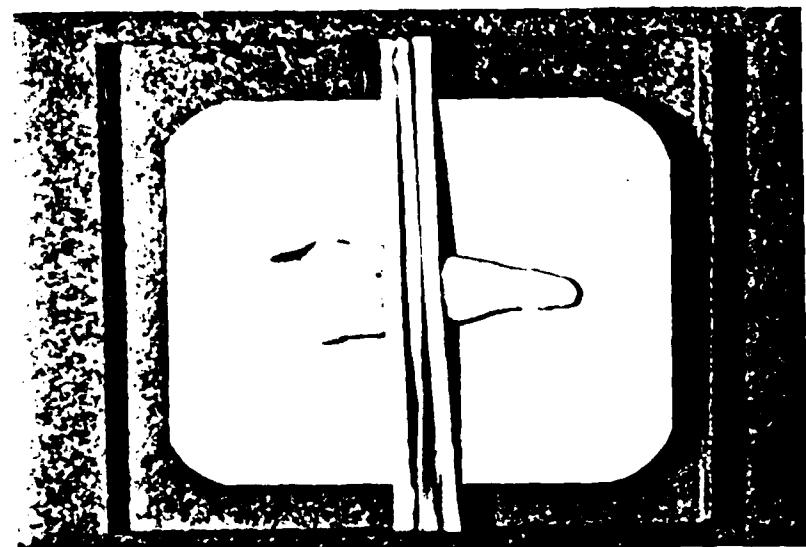
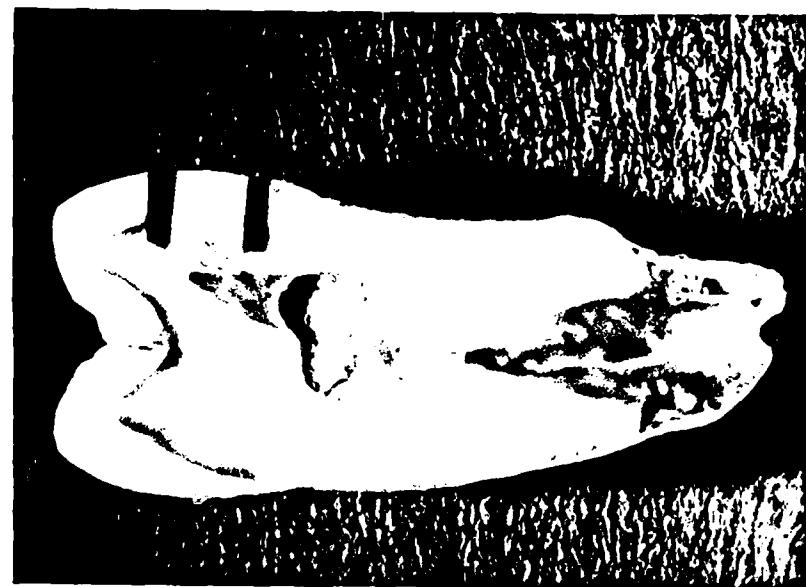


FIGURE 7. Protected film and specimen. The plexiglass, film, and specimen were wrapped in tin foil.

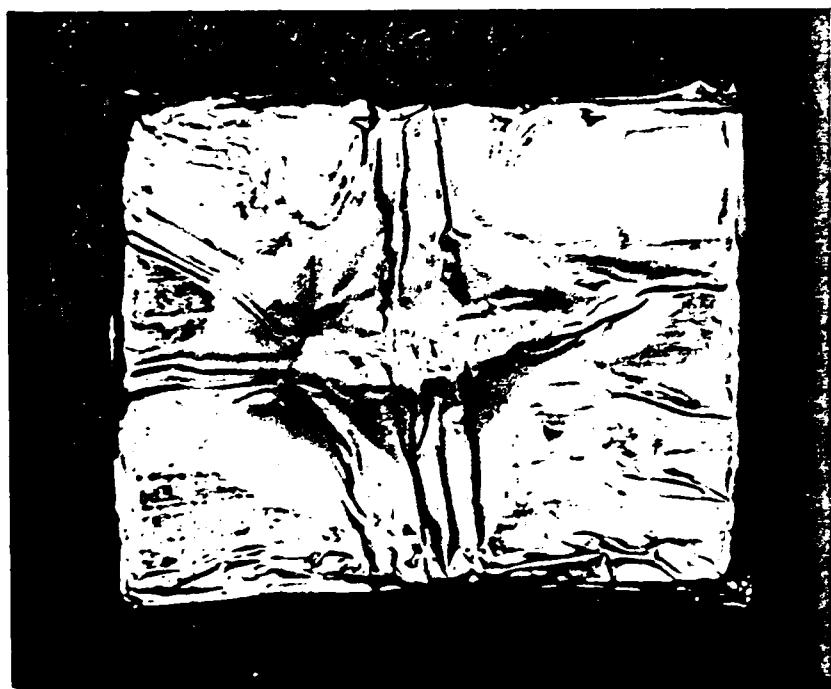


FIGURE 8. Standard for restorative materials.

One set of four autoradiographs served as the standard against which the extent of penetration of the radioisotope at the margins of the restorative materials was scored. Each autoradiograph was evaluated as to no, slight, moderate, or severe penetration of the radioisotope and assigned the appropriate numerical value.

RESTORATIVE MATERIAL LEAKAGE STANDARDS

A      B      C      D



FIGURE 9. Standard for temporary cements.

A second set of four autoradiographs served as the standard against which penetration of radioisotope at the margins of the temporary cements was compared. Penetration was graded as no, slight, moderate, or severe, and assigned the appropriate numerical value.

TEMPORARY CEMENT LEAKAGE STANDARDS

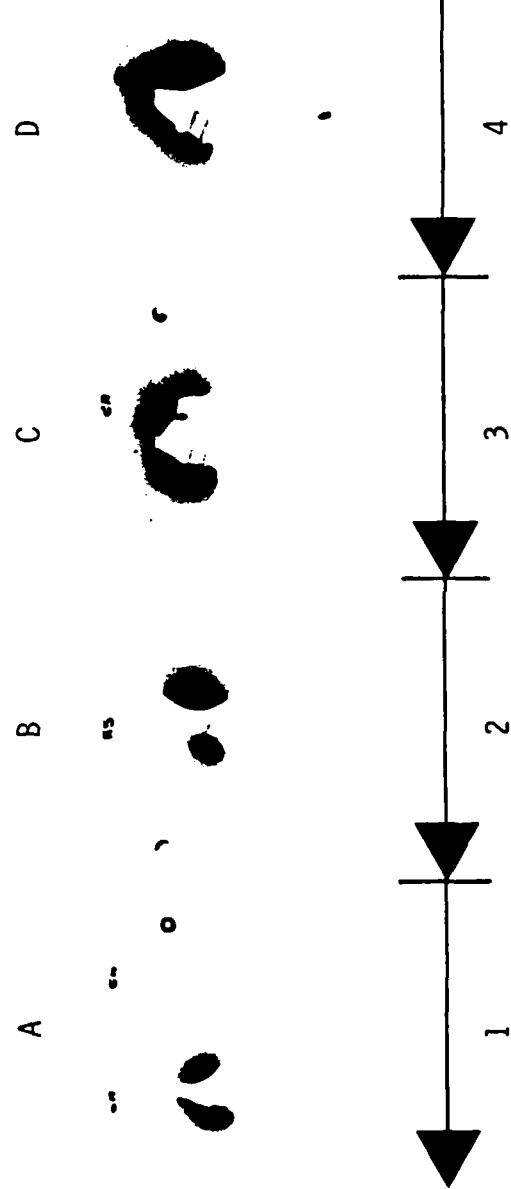


FIGURE 10. Slump tests performed on the three temporary cement mixes:

1. Thick mix
2. Medium mix
3. Thin mix

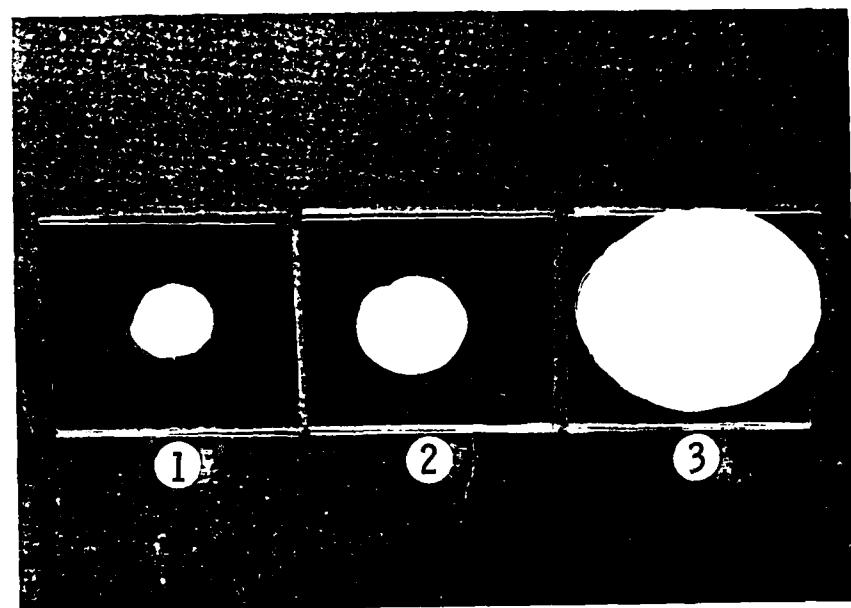


FIGURE 11. Microleakage of temporary cement mixes,  
ridit values, leakage categories and  
test groups.

TEMPORARY CEMENT MIXES  
( Microleakage )

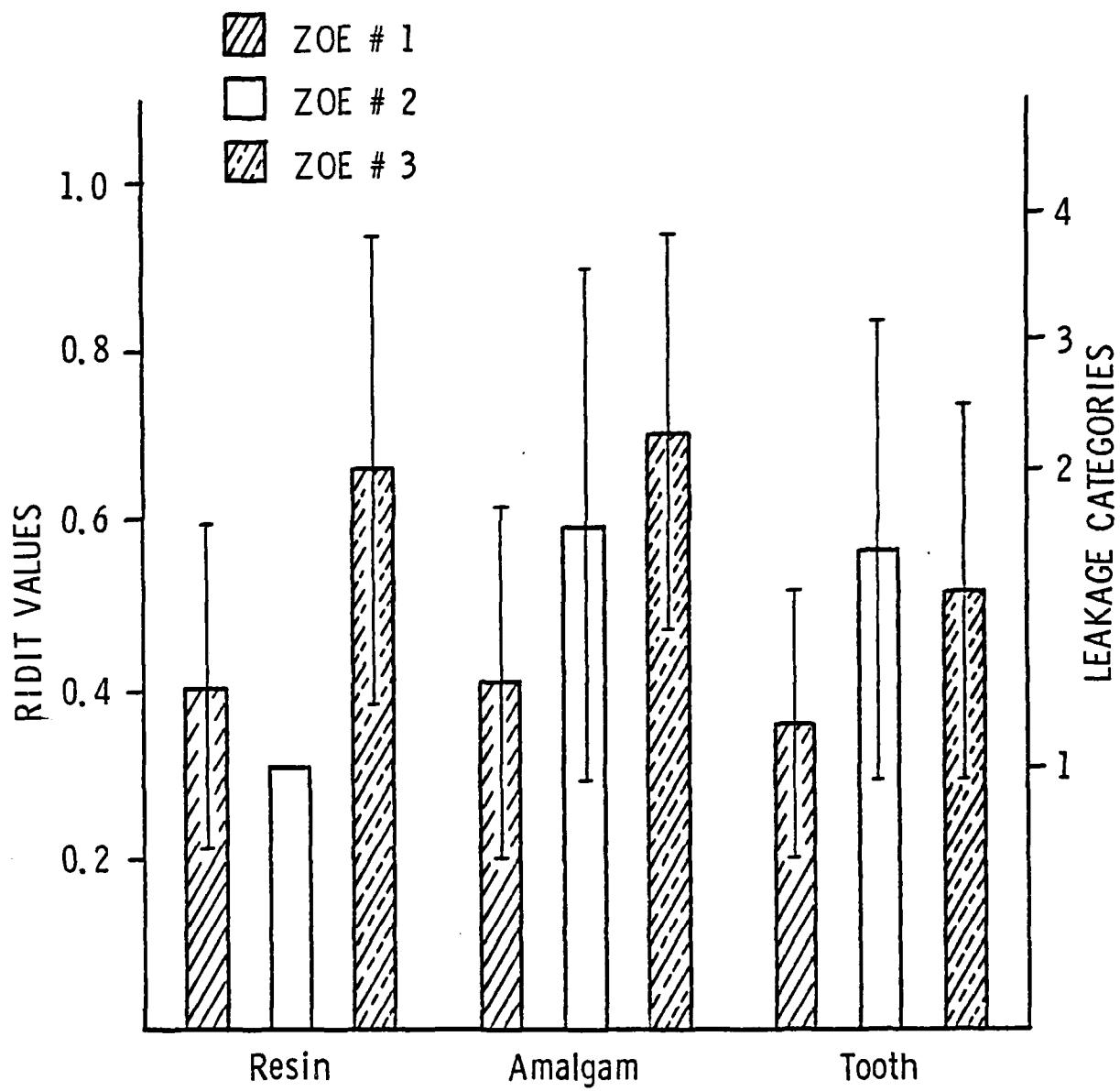


FIGURE 12. Microleakage of restorative materials,  
ridit values, leakage categories and  
test groups.

RESTORATIVE MATERIALS  
( Microleakage )

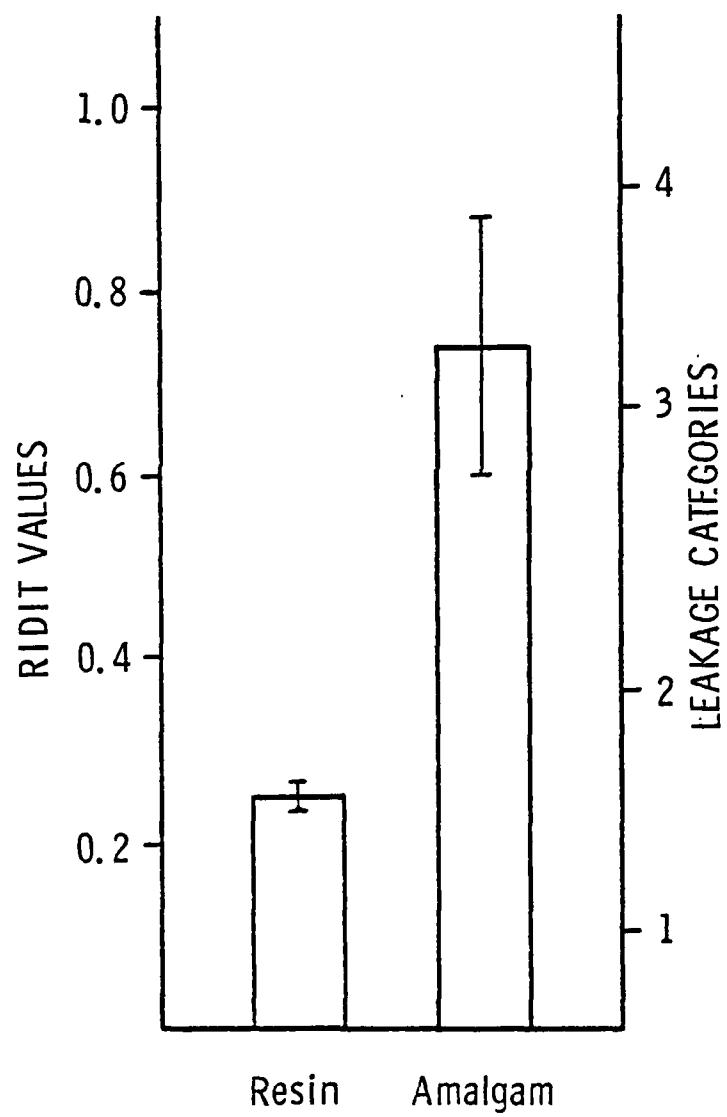


TABLE I

Powder/Liquid Ratios for ZOE Mixes and Average Diameters  
for the Slump Test

GROUP	RATIO	SLUMP TEST AVERAGE DIAMETERS
ZOE Mix #1	1.00 gm powder/0.2 ml eugenol	14 mm
ZOE Mix #2	0.75 gm powder/0.2 ml eugenol	22 mm
ZOE Mix #3	0.50 gm powder/0.2 ml eugenol	50 mm

TABLE II

## Evaluator Correlation

## Intraevaluator Correlation

Correlation was tested using Pearson's Correlation Coefficient.

READINGS	EVALUATOR A	EVALUATOR B
#1 - #2	.91	.87
#1 - #3	.77	.87
#2 - #3	.87	1.00

## Interevaluator Correlation

Evaluators A & B .87

Correlation is demonstrated at the .01 level of significance.

TABLE III

Combined Tabulation of Average of Film Readings by Group

Ridit Analysis of Microleakage

Temporary Restorative Materials

ZOE Mixes	Material	Classification of Leakage							Total
		Ridit Mean	S.D.	#1	#2	#3	#4		
#1	Amalgam	.412	.213	8	0	2	0	10	
	Amalgam	.593	.303	5	0	3	2	10	
	Amalgam	.703	.235	2	1	5	1	9 *	
<hr/>									
#2	Resin	.310	.000	10	0	0	0	10	
	Resin	.406	.193	7	1	1	0	9 *	
	Resin	.664	.280	3	1	3	2	9 *	
<hr/>									
#1	Tooth	.361	.160	9	0	1	0	10	
	Tooth	.517	.225	5	3	2	0	10	
	Tooth	.562	.276	5	1	3	1	10	
<hr/>									
		TOTAL:		54	7	20	6	87	
<hr/>									
RIDIT VALUE: .310 .661 .816 .966									

Mixes connected by a vertical line are not significantly different.

\* One tooth in this group was lost to cracking.

TABLE IV

Tabulation of Ridit Means and Standard Deviations for the Experimental Groups

Ridit Means Placed in Increasing Order of Leakage

GROUP	RIDIT MEAN	S.D.
ZOE #2 - Resin	.310	.000
ZOE #1 - Tooth	.361	.160
ZOE #1 - Resin	.406	.193
ZOE #1 - Amalgam	.412	.213
ZOE #3 - Tooth	.517	.225
ZOE #2 - Tooth	.562	.276
ZOE #2 - Amalgam	.593	.303
ZOE #3 - Resin	.664	.280
ZOE #3 - Amalgam	.703	.235

GROUP	RIDIT MEAN	S.D.
Resin	.254	.017
Amalgam	.740	.143

TABLE V

Combined Tabulation of Average of Film Readings by Group

## Ridit Analysis of Microleakage

## Restorative Materials

CLASSIFICATION OF LEAKAGE

GROUP	#1	#2	#3	#4	Total	Ridit Mean	S.D.
Resin	17	21	0	0	38	.254	.017
Amalgam	0	2	24	13	39	.740	.143
TOTAL:	17	23	24	13	77		
RIDIT VALUE	.110	.370	.675	.916			

## DISCUSSION

Marginal leakage at the interface of restorative material and tooth structure has captured the interest of dentists for years. The search for restorative materials that decrease leakage is a continual process that involves both permanent and temporary restorative materials. The specific requirements of materials differ depending on their intended usage. A prime consideration in selecting a temporary restorative material for use in endodontics is its ability to seal the margins of the access preparation, thereby preventing the exchange of fluid between the root canal system and the mouth. Unless this ingress of bacteria and its byproducts can be prevented, multiple appointment endodontic treatment may not be feasible and single appointment endodontics may be the only alternative.

In vitro marginal leakage studies have routinely investigated the marginal leakage of temporary restorative materials. Numerous reports indicate that all temporary restorative materials exhibit some degree of leakage. These studies show that ZOE generally provides a good seal. More recently Orahood showed that ZOE was very erratic with respect to its ability to provide an effective marginal seal when used to restore simulated access

8,20-22

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openings placed in amalgam or composite resin. Consequently a search was conducted to determine what effects varying the powder/liquid ratio would have on the sealing ability of ZOE. Using a standardized mixing time, as suggested by Orahood, three different powder/liquid ratios of ZOE were tested for this investigation.

Although the usual endodontic access preparation is placed on the lingual of anterior teeth, and occlusal surface of posterior teeth, the facial surface was chosen for this study. A previous in vitro study<sup>60</sup> of marginal leakage using extracted human teeth found more marginal leakage related to developmental grooves and fissures on the lingual and occlusal surfaces than on the facial surfaces. Only anterior and bicuspid teeth with intact facial surfaces were selected for the present research. Molar teeth were not used because the developmental grooves on the buccal surface may allow penetration of the radioisotope which is not related to marginal leakage. In addition, reliable data is more difficult to obtain on molars because of the difficulty<sup>45</sup> in sealing the apices of multirooted teeth from Ca isotope penetration.

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With one exception, all previous studies of the marginal sealing ability of temporary restorative cements using human extracted teeth have tested the cement-tooth interface. This present study tested both the margin at the

cement-restoration interface that is created when endodontic therapy is initiated on a previously restored tooth, and the cement-tooth interface of a tooth without a previous restoration. Amalgam and composite resin were selected as the restorative materials based upon the frequency that these restorations are placed in the mouth compared to other restorations.

Several different methods are available to test the marginal sealing ability of restorative materials and have been discussed at length in the literature review. Ca has been used for many years to assess the in vitro marginal leakage of dental restorative materials. This method, 59 standardized by Swartz and repeated by many investigators since, was used with only slight modifications to accommodate the testing of the temporary restorative cement under investigation. 45 Although Ca is of smaller size than even the smallest bacteria, it has not been proven that the irritants and toxins which we are attempting to seal out of the root canal system with the temporary restorations are as small as 45 or smaller than the Ca ion. With this in mind, testing 45 marginal leakage by  $\text{CaCl}_2$  penetration may be more realistic than testing by bacterial penetration. 45

Evaluating the autoradiographs in Ca studies is difficult and based almost entirely on subjective observation. Two examiners, using the same set of standard

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IN VITRO STUDY COMPARING THE MARGINAL LEAKAGE OF  
DIFFERENT POWDER/LIQUID RATIOS OF ZOE(U) AIR FORCE INST  
OF TECH WRIGHT-PATTERSON AFB OH D J ZENT 1985

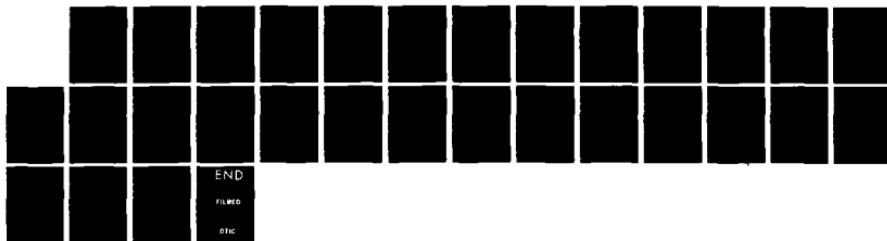
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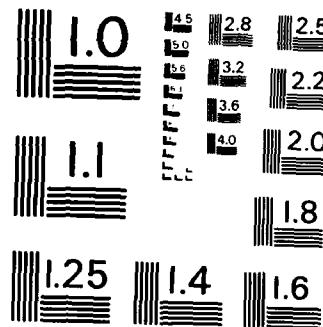
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MICROCOPY RESOLUTION TEST CHART  
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autoradiographs (Figures 8 & 9), assigned a numerical value (Table III) to each radiograph.

The depth of the axial wall of the cavity preparations for the restorative materials was standardized to no more than 3.0 mm. This allowed adequate depth of cement-restorative material interface for testing of isotope penetration. The degree of marginal leakage observed represented a section of each restoration examined. The isotope penetration was evaluated after subjecting the specimens to 2500 thermocycles and an aqueous solution of  $45\text{CaCl}_2$  for two hours. Increased thermal stress could have resulted in a different amount or pattern of leakage.

The simulated access cavity preparation made in the restorative material was always placed completely through the material into dentin. This was done to simulate a clinical situation in which an access cavity is placed through a restoration when making entrance into the pulp chamber. This permitted investigation of the influence of the restoration-tooth interface and the cement-restoration interface. The cement-tooth interface was also examined in specimens which had the access cavity placed in non-restored teeth. In a previous study, using radioisotope generated autoradiographs, it was found that a cotton pellet, which is commonly put in the access cavity prior to temporary cement placement, retained moisture and adversely affected the

quality of the autoradiographs. Therefore, the temporary cement was condensed against the more rigid tooth instead of the soft surface provided by a cotton pellet in a clinical situation. This could have improved the cement-wall adaptation and the marginal seal.

49,67

Previous studies indicate that subjecting extracted teeth to thermocycling with a 40 C temperature differential provides a differential comparable to what occurs when hot and cold foods are ingested in vivo.

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McCurdy used isotopes both in vivo and in vitro to study the leakage of coronal restorations. In comparing his results, he stated that the in vitro laboratory Ca leakage studies are a reliable means of predicting the in vivo sealing ability of a restorative material.

The amalgam and composite resin restorations were completed two weeks prior to the placement of the temporary cement mixes in the simulated access cavity preparations. This interval was selected because endodontic access is rarely made through a just completed restoration and for the purpose of uniformity in this study.

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Both Marosky and Orahood referred to manipulative problems that arise whenever dental materials are tested and compared. The investigator may be more familiar with one of the materials, or he may have an unfavorable attitude towards another. These situations can alter the results.

The different ways in which investigators handle materials in a particular study make comparison between separate tests difficult.

Many pilot studies were performed during this investigation to allow the investigator to become familiar with the ZOE cement to be used and to standardize the mixes and placement technique. The investigator performed all of the material manipulations and the conditions were kept constant for each different test group. The setting times and flow were affected by the powder to liquid ratio as both working time and the flow, as indicated by the slump tests, were inversely proportional to the amount of powder in each mix. The thin mix showed increased flow but it lacked body, and placement was difficult. The medium mix was stiffer and was easier to place. The thick mix had both good body and stiffness and could easily be condensed into the access cavity. The thick mix set the quickest but still allowed sufficient working time to restore the access cavity. All specimens were allowed to bench set for three minutes before being placed in a container of tap water held at 37° C for two weeks. This three minute setting time was within the suggested range of time recommended by the manufacturer for the manipulation and set of ZOE. The two week storage period prior to leakage testing simulated an acceptable inter-  
2 appointment period as described by Weine.

Only in one instance was there a statistical difference between mixes ( $p < .01$ ) and that favored the thick and medium mixes when placed in simulated endodontic access cavities. Overall, the lowest leakage was recorded with the thick temporary restorative mix placed in all three types of access preparation. Nevertheless, the overall results suggest a trend that shows decreased leakage with increased powder/liquid ratio within the range employed here. The handling characteristics and improved seal obtained with the thick mix make it the ratio of choice when restoring endodontic access preparations with ZOE temporary cement. Further investigation using a larger number of specimens is needed to determine the best sealing powder/liquid ratio of ZOE.

The observation that amalgam showed significantly greater marginal leakage than composite resin may be explained by the properties of amalgam previously noted. The high copper amalgam, with its reduced gamma two phase, 63,66,83 is less subject to corrosion. Since numerous studies have reported decreased marginal leakage due to corrosion in aged amalgam restorations, a coat of cavity varnish was placed to decrease the marginal leakage which occurs prior to this corrosion. It is also possible that the observed marginal leakage was due to operator manipulation of the material.

Although this study attempted to simulate a clinical situation as nearly as possible, material hardness or resistance to abrasion was not compared. This would be an important consideration clinically, especially in posterior teeth. It is possible that a double seal would aid in resistance to occlusal forces and abrasion. But to date there are no studies which demonstrate the best combinations of cements which would provide both resistance to occlusal forces and an adequate marginal seal. Further investigation concerning possible variation in material hardness and resistance to abrasion is warranted if an improved sealing mix is obtained by varying the ZOE powder/liquid ratio.

## **SUMMARY AND CONCLUSIONS**

Canal asepsis is a basic principle of the endodontic triad. Since multiple appointments are a widely accepted form of endodontic treatment, the ability to seal the margins of an access preparation with a temporary restorative material has become an important consideration.

Previous investigations have compared the sealing ability of interappointment temporary restorations at the interface of cement and tooth structure. This study assessed the marginal leakage of three powder/liquid ratios of a temporary cement (ZOE), which was inserted into simulated endodontic access preparations placed in tooth structure as well as in amalgam and composite resin.

Class V cavity preparations with a 90° cavosurface angle were made in 110 anterior and bicuspid extracted human teeth. Thirty of these teeth were set aside to await later placement of the temporary mixes. Forty of the teeth were restored with amalgam and the remaining 40 with composite resin. Ten each from the amalgam and composite resin group served as controls. The amalgam was manipulated according to manufacturer's recommendations, then placed in cavity preparations coated with two coats of cavity varnish. Composite resin manipulated according to manufacturer's

recommendations was placed in a resin primed cavity preparation with beveled and etched enamel margins. All specimens were aged two weeks before the temporary cement was placed.

All 30 experimental teeth in each of the three groups had simulated access preparations placed into tooth structure or through the restorative material. In each group, 10 teeth were then restored with a thin ZOE mix, 10 with a medium mix, and 10 with a thick mix. All specimens were stored in tap water at 37° C except for necessary manipulation. All were subjected to the stress of 2500 thermocycles between two water baths with a temperature differential of 40° C and 30-second immersion time in each bath. Each cement mix was tested for marginal leakage two weeks after placement. Marginal penetration was assessed using radioactive <sup>45</sup>Ca generated autoradiographs. Each autoradiograph was given a leakage classification under multiple, coded, blind conditions by two evaluators.

Statistical analysis using ridit mean and standard deviation for each experimental group was performed with multiple comparisons using the Neuman-Keul Test and analysis of variance to determine significant differences ( $p < .05$ ) in leakage values of the groups.

Comparing the three temporary cement mixes, the best seal was observed when the access preparations were placed

through composite resin and restored with the medium mix (ZOE #2). The most leakage was observed when the access preparations were placed through amalgam and restored with the thin mix (ZOE #3). There was a significant difference in marginal leakage of the thick (ZOE #1) and medium (ZOE #2) mixes placed in composite resin when compared to the thin mix (ZOE #3) ( $p < .01$ ). There was no significant difference between any of the mixes placed in amalgam or between the mixes placed in tooth structure ( $p < .05$ ).

Comparing the two restorative materials, amalgam and composite resin, amalgam demonstrated significantly greater marginal leakage than the beveled, etched, resin-primed composite resin ( $p < .01$ ).

## APPENDIXES

## APPENDIX A

Sample computation of evaluator correlation using  
Pearson's Correlation Coefficient

Twenty-five samples selected randomly from each film reading and each evaluator were used for the correlation evaluation. The samples from two different film readings were tabulated and the values obtained were utilized in the formula for computation of Pearson's correlation coefficient.

X	Y	N	XN	YN	$X^2$	$Y^2$	$XYN$
1	1	15	15	15	15	15	15
2	2	3	6	6	12	12	12
3	3	2	6	6	18	18	18
4	4						
1	2	3	3	6	3	12	6
1	3	1	1	3	1	9	3
4	3	1	4	3	16	9	12
		25	35	39	60	75	66

$$r^2_{xy} = \frac{[n \cdot xy_n - (xn)(yn)]^2}{[n \cdot x^2_n - (xn)^2][n \cdot y^2_n - (yn)^2]}$$

$$= \frac{[25(66) - (35)(39)]^2}{[25(60) - (35)^2][25(75) - (39)^2]}$$

$$= .834$$

$$r = .913$$

## APPENDIX B

## Sample Computation of Ridit Value

## 1. Calculation of Ridit Value #1

$$\begin{aligned} \text{R.V. } \#1 &= \frac{0.5(\text{Total Classification } \#1)}{\text{Grand Total}} \\ &= \frac{0.5(54)}{87} \\ &= 0.310 \end{aligned}$$

## 2. Calculation of Ridit Value #2

$$\begin{aligned} \text{R.V. } \#2 &= \frac{(\text{T.C. } \#1) + 0.5 (\text{T.C. } \#2)}{\text{Grand Total}} \\ &= \frac{54 + 0.5 (7)}{87} \\ &= 0.661 \end{aligned}$$

## 3. Calculation of Ridit Value #3

$$\begin{aligned} \text{R.V. } \#3 &= \frac{(\text{T.C. } \#1) + (\text{T.C. } \#2) + 0.5 (\text{T.C. } \#3)}{\text{Grand Total}} \\ &= \frac{54 + 7 + 0.5 (20)}{87} \\ &= 0.816 \end{aligned}$$

## 4. Calculation of Ridit Value #4

$$\begin{aligned} \text{R.V. } \#4 &= \frac{(\text{T.C. } \#1) + (\text{T.C. } \#2) + (\text{T.C. } \#3) + 0.5 (\text{T.C. } \#4)}{\text{Grand Total}} \\ &= \frac{54 + 7 + 20 + 0.5 (6)}{40} \\ &= 0.966 \end{aligned}$$

## APPENDIX C

## Calculation of Ridit Means

$$\text{Ridit Mean} = \frac{(\#1_g)(R.V. \#1) + (\#2_g)(R.V. \#2) + (\#3_g)(R.V. \#3) + (\#4_g)(R.V. \#4)}{N}$$

g = value from a specific group

N = total film readings of a specific group

Example: Ridit mean of ZOE #1/Amalgam

$$\text{Ridit mean} = \frac{8 (0.310) + 0 (0.661) + 2 (0.816) + 0 (0.966)}{10}$$
$$= 0.412$$

## APPENDIX D

## Calculation of Standard Deviations (S.D.)

$$S.D. = \frac{(\#1_g)(R.V.\#1-X_g)^2 + (\#2_g)(R.V.\#2-X_g)^2 + (\#3_g)(R.V.\#3-X_g)^2 + (\#4_g)(R.V.\#4-X_g)^2}{N-1}$$

$\bar{X}$  = Ridit Mean

Example: ZOE #1/Amalgam

$$\frac{8 (0.310 - 0.412)^2 + 0 (0.661 - 0.412)^2 + 2 (0.816 - 0.412)^2 + 0 (0.661 - 0.412)^2}{10 - 1}$$

$$= 0.045$$

$$S.D. = 0.213$$

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ABSTRACT

IN VITRO STUDY COMPARING THE MARGINAL LEAKAGE OF DIFFERENT  
POWDER/LIQUID RATIOS OF ZOE

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This study assessed the marginal leakage of three powder/liquid ratios of a temporary cement (ZOE), which was inserted into simulated endodontic access preparations placed in tooth structure, as well as in amalgam and composite resin.

Class V cavity preparations with a 90° cavosurface angle were made in 110 anterior and bicuspid extracted human teeth. Thirty of these teeth were set aside to await placement of the temporary mixes. Forty of the teeth were restored with amalgam and the remaining 40 with composite resin. Ten each from the amalgam and composite resin groups served as controls. All specimens were aged two weeks before the temporary cement was placed.

All 30 experimental teeth in each of the three groups had simulated access preparations placed into tooth structure or through the restorative material. In each

group, 10 teeth were then restored with a thin ZOE mix, 10 with a medium mix, and 10 with a thick mix.

All specimens were stored in tap water at 37° C except for necessary manipulation. All were subjected to the stress of 2500 thermocycles between two water baths with a temperature differential of 40° C and 30-second immersion time in each bath. Each cement mix was tested for marginal leakage two weeks after placement. Marginal penetration was assessed using radioactive <sup>45</sup>Ca generated autoradiographs. Each autoradiograph was given a leakage classification under multiple, coded, blind conditions by two evaluators.

Statistical analysis using ridit mean and standard deviation for each experimental group was performed with multiple comparisons using the Neuman-Keul Test and analysis of variance to determine significant differences ( $p<.05$ ) in leakage values. There was a significant difference in marginal leakage of the thick and medium mixes placed in composite resin when compared to the thin mix ( $p<.01$ ). Of the restorative material used, amalgam demonstrated significantly greater marginal leakage than the composite resin ( $p<.01$ ).

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